# COOK INLET SUBSISTENCE CONSUMPTION ASSESSMENT OF THE SELDOVIA, PORT GRAHAM, NANWALEK, AND TYONEK TRIBES OF COOK INLET, AK

## Phase II: Contaminant testing of Sockeye Salmon

## Quality Assurance Project Plan

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## 1.0 ACKNOWLEDGEMENTS

This project is being undertaken by the Seldovia Village Tribe (SVT)'s Environmental Staff through an Environmental Protection Agency (EPA) Indian General Assistance Program (IGAP)

Unmet Needs Grant. This project is part of a 2nd phase of a subsistence consumption assessment of Cook Inlet tribes (Seldovia, Port Graham, Nanwalek, and Tyonek). The 1st phase of this assessment was a survey of tribal members to determine consumption rates of subsistence foods (mainly fish and shellfish). This survey took place between November 2011 and September 2012. The 2nd phase of this assessment is tissue sampling of priority subsistence foods for contaminants. SVT was funded to conduct tissue sampling of sockeye salmon within Cook Inlet during the summer of 2014. This will be a collaborative project amongst the four aforementioned tribes, the Alaska Department of Environmental Conservation (ADEC) through their Fish Tissue Testing (i.e. Fish Monitoring) Program, and EPA. We sincerely wish to thank EPA for funding this project, ADEC for providing free laboratory and shipping services, and our partner tribes for assisting with this project.

**2.0 OBJECTIVES**

The objective of this project is to protect and enhance the health of Cook Inlet tribal members by collecting data on contaminants present in priority fish species eaten by tribal members (specifically sockeye salmon). Specific goals include:

1) The analyses of whole body samples of sockeye salmon collected from Cook Inlet to determine the potential for human health and environmental effects associated with levels of chemical contaminants

**Commented [KL1]:** What parts of the fish are consumed? Usually fillet tissues are analyzed. If the desire is to get whole body concentrations for ecological risk assessment, it might be helpful to have both whole body and fillet samples analyzed to determine contaminant relationships between the two. Another consideration might be sampling organism at different life stages if environmental risk assessment is being considered. Levels of contaminants in juvenile salmonids and/or eggs are of potential concern.

I'm curious as to why only salmon and not other species are being sampled. Organisms with smaller home ranges are likely to be more impacted by regional contamination than salmon, which spend a significant portion of their lives in the open ocean.

2) To establish a more comprehensive database of contaminant concentrations within Cook Inlet for evaluation and use in establishing or revising water quality standards, in the issuance or removal of human health fish consumption advisories, and in environmental impact assessments.

This QAPP is designed to ensure that all fish tissue sample analytical results are of consistent, high quality so that the best information is made available to evaluate and protect traditional resources of Cook Inlet tribal members.

**3.0 BACKGROUND**

Cook Inlet stretches 180 miles (290 km) from the Gulf of Alaska to Anchorage in south-central Alaska. This large tidal estuary covers about 100,000 km² of southern Alaska, east of the Aleutian Range and south of the Alaska Range. At least 150 rivers and streams empty into Cook Inlet. For thousands of years, native Alaskans have relied on the rich diversity and abundance of animals and plants residing in Cook Inlet as traditional foods. Development and oil and gas activities occurring in Cook Inlet have raised great concerns over contaminants in traditional foods harvested within Cook Inlet and the risk these contaminants pose to human health.

*PAST EVALUATIONS*

**Commented [KL2]:** What this section is missing is a discussion of what data are needed to evaluate human and ecological risks, where these results fit into overall data needs, what further work needs to be done, and how this sampling effort will fit into a broader sampling plan.

Previous investigations by federal and state agencies have identified metals, pesticides, polychlorinated biphenyls (PCB), polycyclic aromatic hydrocarbons (PAH) and dioxin compounds in traditionally eaten foods from Cook Inlet. Contaminant data from tissue sampling of fish and

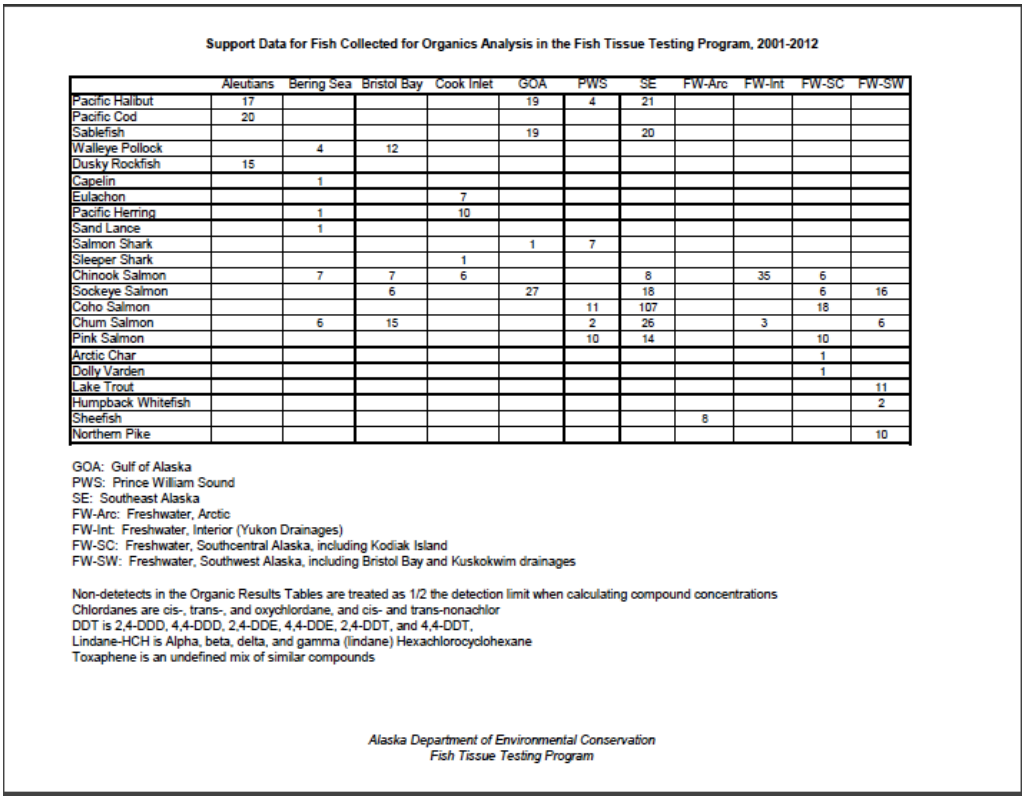
shellfish within Cook Inlet has been previously collected through studies undertaken by multiple agencies and organizations.

These studies/projects include:

Commented [KL3]: Maps showing areas that were studied, ideally including sampling locations, would make it much easier for the reader to understand the coverage of existing data. This is particularly true for individuals who are not familiar with the geography of the Seldovia Region.

1. Fish Tissue Testing Program (i.e. Fish Monitoring Program) – ADEC:

Supported by funding from the EPA, National Oceanic and Atmospheric Administration (NOAA) and Bureau of Ocean Energy Management, Regulation, and Enforcement (BOEMRE), contaminant data for salmon (all five species), halibut, pacific cod, sablefish, black rockfish, sheefish, lingcod, pollock as well as other marine and fresh water species throughout Alaska have been collected for trace metals (methyl mercury, total mercury, selenium, copper, lead, cadmium) and organic contaminants. Most of the data specific to Cook Inlet was collected between 2001 and 2010. More information about this program can be found at http://www.dec.state.ak.us/ADEC EH/vet/fish.htm.



**Support Data for Fish Collected for Organics Analysis in the Fish Tissue Testing Program, 2001-2012**

| | Aleutians | Bering Sea | Bristol Bay | Cook Inlet | GOA | PWS | SE | FW-Arc | FW-Int | FW-SC | FW-SW |
|---|---|---|---|---|---|---|---|---|---|---|---|
| Pacific Halibut | 17 | | | | 19 | 4 | 21 | | | | |
| Pacific Cod | 20 | | | | | | | | | | |
| Sablefish | | | | | 19 | | 20 | | | | |
| Walleye Pollock | | 4 | 12 | | | | | | | | |
| Dusky Rockfish | 15 | | | | | | | | | | |
| Capelin | | 1 | | | | | | | | | |
| Eulachon | | | | 7 | | | | | | | |
| Pacific Herring | | 1 | | 10 | | | | | | | |
| Sand Lance | | 1 | | | | | | | | | |
| Salmon Shark | | | | | 1 | 7 | | | | | |
| Sleeper Shark | | | | 1 | | | | | | | |
| Chinook Salmon | | 7 | 7 | 6 | | | 8 | | 35 | 6 | |
| Sockeye Salmon | | | 6 | | 27 | | 18 | | | 6 | 16 |
| Coho Salmon | | | | | | 11 | 107 | | | 18 | |
| Chum Salmon | | 6 | 15 | | | 2 | 26 | | 3 | | 6 |
| Pink Salmon | | | | | | 10 | 14 | | | 10 | |
| Arctic Char | | | | | | | | | | 1 | |
| Dolly Varden | | | | | | | | | | 1 | |
| Lake Trout | | | | | | | | | | | 11 |
| Humpback Whitefish | | | | | | | | | | | 2 |
| Sheefish | | | | | | | | 8 | | | |
| Northern Pike | | | | | | | | | | | 10 |

GOA: Gulf of Alaska
PWS: Prince William Sound
SE: Southeast Alaska
FW-Arc: Freshwater, Arctic
FW-Int: Freshwater, Interior (Yukon Drainages)
FW-SC: Freshwater, Southcentral Alaska, including Kodiak Island
FW-SW: Freshwater, Southwest Alaska, including Bristol Bay and Kuskokwim drainages

Non-detetects in the Organic Results Tables are treated as 1/2 the detection limit when calculating compound concentrations
Chlordanes are cis-, trans-, and oxychlordane, and cis- and trans-nonachlor
DDT is 2,4-DDD, 4,4-DDD, 2,4-DDE, 4,4-DDE, 2,4-DDT, and 4,4-DDT,
Lindane-HCH is Alpha, beta, delta, and gamma (lindane) Hexachlorocyclohexane
Toxaphene is an undefined mix of similar compounds

*Alaska Department of Environmental Conservation*
*Fish Tissue Testing Program*

In terms of Cook Inlet data, what ADEC presently has contaminant information on (as of December 2013) is:

| Heavy Metals: | | Organics: | |
|---|---|---|---|
| Species/Taxa | Years of Data | Species/Taxa | Years of Data |
| Clams | 1996-2001 | | |
| Sleeper Shark | 2013 | Sleeper Shark | 2013 |
| Pacific Cod | 2001-2009 | Pacific Cod | 2010 |
| Dolly Varden | 2008 | | |
| Eulachon | 2009 | Eulachon | 2010 |
| Grayling | 2008 | | |
| Halibut | 2002-2007 | | |
| Pacific Herring | 2008-2010 | Pacific Herring | 2008 |
| Lingcod | 2002 and 2010 | | |
| Walleye Pollock | 2002 and 2009 | | |
| Rockfish | 2007 | | |
| Chinook Salmon | 2001 and 2006 | Chinook Salmon | 2002 |
| Chum Salmon | 2002 | | |
| Pink Salmon | 2002 | | |
| Sockeye Salmon | 2002-2003 | | |
| Coho Salmon | 2002-2006 | | |
| Spiny Dogfish | 2001-2002 | | |
| Rainbow Trout | 2009 | | |

| Sample Type | Tissue | Region | N for arsenic, cadmium, lead | N for copper | N for selenium | N for mercury |
|---|---|---|---|---|---|---|
| Pacific Halibut | Fillet | Homer | 56 | 48 | 56 | 56 |
| Pacific Cod | Fillet | Homer | 45 | 34 | 45 | 45 |
| Lingcod | Fillet | Homer | 17 | 13 | 17 | 17 |
| Kelp Greenling | Whole | Homer | 5 | 5 | 5 | 5 |
| Walleye Pollock | Fillet | Homer | 14 | 3 | 14 | 14 |
| Black Rockfish | Fillet | Homer | 2 | 0 | 2 | 2 |
| Dusky Rockfish | Fillet | Homer | 3 | 2 | 3 | 3 |
| Rougheye Rockfish | Fillet | Homer | 17 | 15 | 17 | 17 |
| Yelloweye Rockfish | Fillet | Homer | 2 | 0 | 2 | 2 |
| Eulachon | Whole Composite | Kenai | 7 | 7 | 7 | 7 |
| Pacific Herring | Whole Composite | Homer | 10 | 10 | 10 | 10 |
| Starry Flounder | Whole | Homer | 1 | 1 | 1 | 1 |
| Southern Rock Sole | Whole | Homer | 1 | 1 | 1 | 1 |
| Sleeper Shark | Fillet | Homer | 1 | 1 | 1 | 1 |
| Spiny Dogfish | Fillet | Homer | 1 | 0 | 0 | 0 |
| | Fillet | Kenai | 1 | 0 | 1 | 0 |
| Chinook Salmon | Fillet | Homer | 6 | 0 | 6 | 6 |
| | Fillet | Kenai | 5 | 0 | 5 | 5 |
| Sockeye Salmon | Fillet | Homer | 6 | 0 | 6 | 6 |
| | Fillet | Kenai | 9 | 0 | 9 | 9 |
| Coho Salmon | Fillet | Homer | 6 | 0 | 6 | 6 |
| | Fillet | Kenai | 10 | 0 | 10 | 10 |
| Chum Salmon | Fillet | Homer | 6 | 0 | 6 | 6 |
| Pink Salmon | Fillet | Homer | 6 | 0 | 6 | 6 |
| Grayling | Fillet | Kenai | 8 | 8 | 8 | 8 |
| Dolly varden | Fillet | Kenai | 6 | 6 | 6 | 6 |
| Northern Pike | Fillet | Kenai | 1 | 1 | 1 | 1 |
| Rainbow Trout | Fillet | Kenai | 2 | 2 | 2 | 2 |
| Butter Clam | Whole Tissue | Homer | 16 | 0 | 0 | 0 |
| Cockle | Whole Tissue | Homer | 4 | 0 | 0 | 0 |
| littleneck clam | Whole Tissue | Homer | 29 | 0 | 0 | 0 |
| Razor Clam | Muscle Tissue | Homer | 21 | 0 | 0 | 0 |
| | Muscle Tissue | Kenai | 3 | 0 | 2 | 2 |
| | Muscle Tissue | West Cook Inlet | 17 | 0 | 0 | 0 |
| Redneck Clam | Whole Tissue | Homer | 6 | 0 | 0 | 0 |
| | Whole Tissue | Kenai | 1 | 0 | 0 | 0 |
| | Whole Tissue | West Cook Inlet | 1 | 0 | 0 | 0 |
| Softshell Clam | Whole Tissue | Homer | 2 | 0 | 0 | 0 |
| Blue Mussel | Whole Tissue | Homer | 47 | 0 | 0 | 0 |
| Pacific Oyster | Whole Tissue | Homer | 56 | 0 | 1 | 1 |
| Bay Scallop | Muscle Tissue | Homer | 9 | 0 | 0 | 0 |

| | | | | | | |
|---|---|---|---|---|---|---|
| Neptunea pribilofensis snail | Whole Tissue | West Cook Inlet | 1 | 0 | 0 | 0 |
| Octopus | Whole Tissue | Homer | 1 | 0 | 0 | 0 |

Typical contaminant levels found in sockeye salmon in Alaskan waters (based on ADEC fish tissue monitoring program data). ND=not detected. Data available at http://dec.alaska.gov/eh/vet/fish.htm:

*PCBs, PBDEs, and pesticides (Concentrations in parts/billion wet weight)*

| Contaminant | Type of sample | Mean ± Std Dev | Median | Range |
|---|---|---|---|---|
| PCBs-Congener 153 | Fillet | .54 ± .46 | .30 | 0.017-2.0 |
| | Whole | .54 ± .29 | .49 | 0.15-1.3 |
| PCBs-total | Fillet | 6.8 ± 5.6 | 4.4 | 0.24-23 |
| | Whole | 7.1 ± 3.5 | 6.5 | 2.2-17 |
| PBDE-Congener 47 | Fillet | .083 ± .16 | .040 | .009-.93 |
| | Whole | .23 ± .33 | .074 | .041-1.2 |
| PBDEs-total | Fillet | .31 ± .35 | .20 | .074-2.1 |
| | Whole | .74 ± .97 | .27 | .13-3.7 |
| Pesticides-sum DDT | Fillet | 7.3 ± 5.9 | 5.0 | .17-22 |
| | Whole | 5.2 ± 2.5 | 4.9 | 1.4-9.7 |
| Pesticides-sum Chlordanes | Fillet | 10 ± 24 | 1.9 | ND-113 |
| | Whole | 1.9 ± .65 | 1.9 | .84-3.2 |
| Pesticides-total Toxaphenes | Fillet | 12 ± 10 | ND | ND-39 |
| | Whole | 16 ± 7.8 | 14 | ND-30 |
| Pesticides-Dieldrin | Fillet | .38 ± .29 | .30 | ND-1.3 |
| | Whole | .30 ± .11 | .27 | .08-.51 |
| Pesticides-Lindane and other hexachlorocyclohexane | Fillet | 1.2 ± 1.2 | .78 | .10-5.2 |
| | Whole | .85 ± .67 | .76 | .06-2.4 |
| Pesticides-hexachlorobenzene | Fillet | 1.2 ± .80 | .96 | .20-3.7 |
| | Whole | 1.4 ± .47 | 1.4 | .46-2.1 |

*Dioxins/Furans (Concentrations in parts/trillion wet weight)*

| Contaminant | Type of sample | Mean ± Std Dev | Median | Range |
|---|---|---|---|---|
| Dioxins/Furans-2,3,7,8-Tetrachloro-dilbenzo-dioxin | Fillet | ND | ND | ND-.088 |
| | Whole | .051 ± .019 | 0.059 | ND-.077 |
| Dioxins/Furans-Sum of 4 to 8 Chlorine | Fillet | 1.4 ± .94 | 1.1 | .27-4.8 |
| | Whole | 1.4 ± .76 | 1.2 | .34-3.2 |

**Commented [KL4]:** Are these toxic equivalents (i.e. the concentration of 2,3,7,8-TCDD that is equivalent to the sum of the toxic equivalents of all dioxins and furans that have 2,3,7,8-TCDD like activity)?

*Heavy Metals (Concentrations in parts/million wet weight)*

| Contaminant | Type of sample | Mean ± Std Dev | Median | Range |
|---|---|---|---|---|

| | | | | |
|---|---|---|---|---|
| Total mercury | Fillet | .038 ± .014 | .038 | ND-.082 |
| | Whole | .031 ± .010 | .033 | .012-.051 |
| Arsenic | Fillet | .27 ± .12 | .26 | ND-.95 |
| | Whole | .28 ± .084 | .27 | .12-.43 |
| Cadmium | Fillet | ND | ND | ND-.070 |
| | Whole | .029 ± .014 | .026 | .011-.061 |
| Chromium | Fillet | ND | ND | ND-.16 |
| | Whole | .20 ± .13 | .21 | .070-.32 |
| Copper | Fillet | .67 ± .21 | .63 | .41-1.5 |
| | Whole | 5.4 ± 2.6 | 6.1 | .84-9.0 |
| Lead | Fillet | ND | ND | ND-.030 |
| | Whole | ND | ND | ND-.044 |
| Nickel | Fillet | ND | ND | ND-.29 |
| | Whole | .22 ± .064 | .23 | .13-.28 |
| Selenium | Fillet | .23 ± .061 | .23 | .090-.46 |
| | Whole | .54 ± .11 | .54 | .30-.70 |

**Commented [KL5]:** Is this total arsenic or inorganic arsenic? Usually only inorganic arsenic is considered to be of toxicologic significance.

2. Assessment of Contaminant Body Burdens and Histopathology of Fish and Shellfish Species Frequently Used for Subsistence Food by Chugach Native Communities (NPRB - Project-1019; July 1, 2010-February 28, 2013):

This project was a collaborative effort amongst the Chugach Regional Resources Commission (CRRC), the Alutiiq Pride Shellfish Hatchery, the NOAA National Status and Trend (NS&T) Program, and the Northwest Fishery Science Center (NWFSC). This study assessed the contaminant status and histopathology condition of two species of salmon (chum and sockeye salmon) and shellfish (cockles and littleneck clams) commonly harvested by natives in the Chugach region. The fish and shellfish were collected from traditional subsistence harvest areas in the vicinity of Nanwalek, Port Graham and Seldovia. Tissue was analyzed for trace metals and residues of organic contaminants routinely monitored by NS&T program, and histologically characterized for the presence, prevalence and severity of tissue pathology, disease, and parasite infection.

3. Evaluation of seafood and plant data collected from Cook Inlet near the native villages of Port Graham, Nanwalek, Seldovia, and Tyonek, Alaska- Agency for Toxic Substances and Disease Registry (ATSDR)-2009:

EPA collected whole fish, mussels/clams, other invertebrates (i.e. snail, chiton, and octopus) and plants from Cook Inlet in 1997. Between June and August 2002, ADEC collected 65 fish (as part of their Fish Tissue Testing Program) that included Pacific cod, chinook salmon, pink salmon, chum salmon, red salmon, silver salmon, pollock, and halibut from lower Cook Inlet. Skinless fillets and halibut roast from 47 fish were analyzed for heavy metals. Fillets from six Chinook salmon were also analyzed for pesticides, dioxins, and polychlorinated biphenyls (PCBs).

4. Cook Inlet Regional Citizens Advisory Council (CIRCAC) Environmental Monitoring Program – 1993, 1996, and 2000:

Beginning in 1993, CIRCAC began a series of preliminary studies to assess impacts of oil and gas operations on Cook Inlet. In 1993 and 1996, total polycyclic aromatic hydrocarbons (PAHs) were measured in mussels and deposit-feeding clams from seven locations in Cook Inlet and one

**Commented [KL6]:** Would be good to see if these data could be used to derive concentrations of carcinogenic polycyclic aromatic hydrocarbons.

location in Shelikof Strait. In 2000, PAH concentrations were measured in 3 razor clams, 2 mussels, and 3 deposit-feeding clams from the east side of upper Cook Inlet; 4 soft shell clams, 1 razor clam, and 2 deposit-feeding clams from the middle of upper Cook Inlet; and 5 deposit-feeding clams, 1 mussel, 2 razor clams, and 1 softshell clam from the west side of upper Cook Inlet.

**Commented [KL7]:** These sample numbers are inadequate to compute upper confidence limits on the mean. Concentration UCLs are the statistic used to compute human health risks.

Contaminants within the water column and sediments of Cook Inlet have also been examined. These contaminants can subsequently influence contaminants found in traditional foods harvested from Cook Inlet waterways. Potential reference sources for these contaminant data are:

1. Hartwell, S.I., Apeti, D., Claflin, L.W., Johnson, W.E. and Kimbrough, K. 2009. Sediment Quality Triad Assessment in Kachemak Bay: Characterization of Soft Bottom Benthic Habitats and Contaminant Bioeffects Assessment. NOAA Technical Memorandum NOS NCCOS 104. 170pp. (NPRB - Project 726; 7/1/2007-10/30/2009)

2. Pollution and Biological Health Assessment of Fjords on Kenai Peninsula, Alaska

NOAA/NCCOS Project Status: This project began in August 2009 and is still ongoing

This study builds on the National Status and Trends (NS&T) bioeffects assessment of the northern side of Kachemak Bay, completed in 2007, and an assessment in the deep central portion of Kachemak Bay, conducted in 2008 in collaboration with the Cook Inlet Regional Citizens Advisory Council (CIRCAC). It is a joint project with the Alaska Department of Environmental Conservation.

A baseline environmental characterization was done of the fjords and embayments along the south shore of Kachemak Bay and the outer Kenai Peninsula using the sediment quality triad approach. The triad includes: sediment chemistry, sediment toxicity, and benthic invertebrate community structure. Concentrations of over 120 organic and metallic contaminants are being analyzed. Sediment toxicity is being assessed using amphipod bioassays with sediment from the abandoned mine sites. Fish and mussels from selected locations are undergoing contaminant body burden analyses.

Joint field operations were completed in 2009 with the assistance of the NOAA Kasitsna Bay Laboratory and the Kachemak Bay Estuarine Research Reserve.

Thus far, they have found that organic contaminants were elevated in the vicinity of Seldovia Harbor.

3. Frenzel, S.A. 2000. Selected Organic Compounds and Trace Elements in Streambed Sediments and Fish Tissues, Cook Inlet Basin, Alaska. U.S. Geological Survey, Water-Resources Investigations Report 00-4004.

4. Henrichs, S.M., Schell, D.M., Borland, T., Howe, T. 2003. Hydrocarbon sources in Kachemak Bay Sediments: Improved Discrimination by Specific Compound $\delta$ 13C Measurements. Institute of Marine Science, School of Fisheries and Ocean Sciences, University of Alaska Fairbanks. Final Report submitted to the NOAA/UNH Cooperative Institute for Coastal and Estuarine Environmental Technology (CICEET).

5. Saupe, S.M., J. Gendron, and D. Dasher. 2005. The Condition of Southcentral Alaska Coastal Bays and Estuaries. A Statistical Summary for the National Coastal Assessment Program Alaska Department of Environmental Conservation, MARCH 15, 2006.

6. Segar, D.A. 1995. Current water quality in Cook Inlet, Alaska, Study. Environment and Natural Resources Institute, University of Alaska Anchorage. Report for U.S. Department of the Interior, Mineral Management Services, OCS Study MMS 95-0009.

7. Kinnetic Laboratories, Inc. 2004. Knik Arm Crossing Preliminary Offshore Water Quality Assessment Technical Memorandum. Report for Knik Arm Bridge and Toll Authority, Alaska Department of Transportation and Public Facilities, Federal Highway Administration, and HDR Alaska, Inc.

*8. SVT – 2005*. Indian General Assistance Program (IGAP) funded work. Data/Results summarized in SVT's summer 2005 Baseline Sampling Report.

In 2005, SVT environmental staff collected water and sediment samples from sites within Seldovia Bay as part of IGAP funded work. Seldovia Bay Sites consisted of sites along transect lines, four benthic sites, four sewage discharge localized sites and three land fill seepage localized sites. The four transects (T1, T2, T3 and T4) were logically placed in Seldovia Bay to represent the outer bay interface with Kachemak Bay (T1), the outer third of the bay (T2), the mid bay (T3) and the inner bay and interface with the flats and bay's primary fresh water sources. Each of these transect lines had three stations located along their axis. Benthic sites (B1, B2, B3 and B4) were also oriented from the outer bay towards the inner bay. These benthic sites where all either defined sinks or localized deeper spots that were likely to contain trapped sediments. The other sites were issue oriented sampling specific sites including the sewage discharge line (SD1, SD2, SD3 and SD4) moving away from and into the bay from the discharge out fall pipe (SD1) and then Dan's Cove which is a small cove with a small stream running out from below an old dump site (DC1, DC2 and DC3).

**Commented [KL8]:** Providing a map to visualize station locations would be most helpful.

The following table shows the actual chemical sampling events in chronological order by date which were sent in to the laboratory, Analytica, for analysis of polycyclic aromatic hydrocarbons (PAH's), volatile organic compounds, and metals (Arsenic, Barium, Cadmium, Chromium, Lead, Selenium, Mercury). Both water and sediment samples were analyzed.

| Lab Sample Number | Client Description | Matrix | Date Sampled | Time Sampled | Date Received |
|---|---|---|---|---|---|
| A0508057-01 | TIS SW | Salt Water | 3-Aug | 9:07 AM | 4-Aug |
| A0508057-02 | TID SW | Salt Water | 3-Aug | 9:30 AM | 4-Aug |
| | | | | | |
| A0508079-01 | T4S | Salt Water | 4-Aug | 10:52 AM | 5-Aug |
| A0508079-02 | B2D | Salt Water | 4-Aug | 9:22 AM | 5-Aug |
| A0508079-03 | B1D | Salt Water | 4-Aug | 9:00 AM | 5-Aug |
| A0508079-04 | B4S | Salt Water | 4-Aug | 10:03 AM | 5-Aug |
| A0508079-05 | B3S | Salt Water | 4-Aug | 9:41 AM | 5-Aug |
| A0508079-06 | B3D | Salt Water | 4-Aug | 9:52 AM | 5-Aug |
| A0508079-07 | B2S | Salt Water | 4-Aug | 9:12 AM | 5-Aug |
| A0508079-08 | B4D | Salt Water | 4-Aug | 10:16 AM | 5-Aug |
| A0508079-09 | T4D | Salt Water | 4-Aug | 10:45 AM | 5-Aug |
| A0508079-10 | B1S | Salt Water | 4-Aug | 8:45 AM | 5-Aug |
| A0508079-11 | DC SW | Salt Water | 4-Aug | 11:09 AM | 5-Aug |
| A0508079-12 | DC FW | Fresh Water | 4-Aug | 11:00 AM | 5-Aug |
| | | | | | |
| A0508097-01A | SD1 Outfall Pipe | Salt Water | 8-Aug | 9:20 AM | 8-Aug |
| A0508097-02A | SD 2 MZ | Salt Water | 8-Aug | 9:07 AM | 8-Aug |
| A0508097-03A | SD 3 | Salt Water | 8-Aug | 10:10 AM | 8-Aug |
| A0508097-04A | SD 4 | Salt Water | 8-Aug | 10:38 AM | 8-Aug |
| A0508097-05A | SD 4 | Sediment | 8-Aug | 10:43 AM | 8-Aug |
| A0508097-06A | SD 1 Outfall Pipe | Sediment | 8-Aug | 9:55 AM | 8-Aug |
| A0508097-07A | SD 3 | Sediment | 8-Aug | 10:22 AM | 8-Aug |
| A0508097-08A | SD 2 | Sediment | 8-Aug | 9:22 AM | 8-Aug |
| | | | | | |
| A0508149-01 | B1 | Sediment | 10-Aug | 9:15 AM | 10-Aug |
| A0508149-02 | SD # 2 | Sediment | 10-Aug | 10:56 AM | 10-Aug |
| A0508149-03 | B3 | Sediment | 10-Aug | 11:26 AM | 10-Aug |
| A0508149-04 | B4 | Sediment | 10-Aug | 11:49 AM | 10-Aug |
| A0508149-05 | DC | Sediment | 10-Aug | 12:10 PM | 10-Aug |
| A0508149-06 | DC2 | Sediment | 10-Aug | 12:35 PM | 10-Aug |
| A0508149-07 | DC3 | Sediment | 10-Aug | 12:42 PM | 10-Aug |
| | | | | | |
| A0508234-01 | B1 Shallow | Salt Water | 18-Aug | 8:55 AM | 19-Aug |
| A0508234-02 | B1 | Sediment | 18-Aug | 9:06 AM | 19-Aug |
| A0508234-03 | B2 | Sediment | 18-Aug | 9:40 AM | 19-Aug |
| A0508234-04 | B3 | Sediment | 18-Aug | 10:40 AM | 19-Aug |
| A0508234-05 | B4 | Sediment | 18-Aug | 11:37 AM | 19-Aug |
| A0508234-06 | DC | Sediment | 18-Aug | 10:57 AM | 19-Aug |
| A0508234-07 | DC2 | Sediment | 18-Aug | 11:55 AM | 19-Aug |
| A0508234-08 | SD SW | Salt Water | 18-Aug | 11:08 AM | 19-Aug |
| A0508234-09 | Trip Blank | Water | 18-Aug | | 19-Aug |
| A0508234-10 | SD | Sediment | 18-Aug | 11:18 AM | 19-Aug |
| A0508234-11 | DC3 | Sediment | 18-Aug | 12:16 PM | 19-Aug |
| | | | | | |
| Sediment Metals | | | | | |
| A508156 | B1 | Sediment | 10-Aug | 9:15 AM | 16-Aug |
| A508156 | B3 | Sediment | 10-Aug | 11:26 AM | 16-Aug |
| A508156 | B4 | Sediment | 10-Aug | 11:49 AM | 16-Aug |
| A508156 | DC | Sediment | 10-Aug | 12:10 PM | 16-Aug |
| | | | | | |
| A508240 | SD | Sediment | 18-Aug | 11:18 AM | 24-Aug |
| A508240 | B2 | Sediment | 18-Aug | 9:40 AM | 24-Aug |

*PRESENT CONCERNS*

Based on existing data, levels of chemicals found in many native foods from Cook Inlet appear to be often at levels that are found in fish from other parts of Alaska or from grocery stores (ATSDR 2009, Apeti et al. 2013, ADEC Fish Monitoring Program data). Additionally, in general, sockeye salmon in Alaska waters appear to have contaminant levels around, or below, those found in salmon within the Columbia River Basin (see below table),

Range of chemical concentrations found in salmon in Columbia River Basin fish tissue samples (whole-body). Data found in US EPA. 2002. Columbia River Basin Fish Contaminant Survey 1996-1998. EPA Report 910-R-02-006:

| | Fall Chinook | | Spring Chinook | | Coho | |
|---|---|---|---|---|---|---|
| Contaminant | ug/kg | ppm | ug/kg | ppm | ug/kg | ppm |

**Commented [KL9]:** What are the major sources of contamination to Cook Inlet? Where are they? Where are harvest areas and how close are they to contaminant sources?

It would be good to present this comparison in greater detail. Bar charts with actual concentrations presented as bar labels would be a much more effective way of presenting this information than attempting to view it in table format.

When comparing PCB concentrations across studies, some consideration should be given to the analytical method. Ideally, results should be based on the same analytical method. Otherwise, it is important to examine adjusting results to reflect differences in analytical results.

| | | | | | | |
|---|---|---|---|---|---|---|
| Arsenic | 610-1000 | .61-1 | 570-1100 | .57-1.1 | 450-560 | .45-.56 |
| Cadmium | 5-10 | .005-.01 | 6-170 | .006-.17 | 19-27 | .019-.027 |
| Copper | 1000-14000 | 1-14 | 1100-2300 | 1.1-2.3 | 720-2400 | .72-2.4 |
| Lead | 11-1200 | .011-1.2 | <10-92 | <.01-.092 | 11-20 | .011-.02 |
| Mercury | <50-200 | <.05-.2 | <71-130 | <.071-.13 | 11-20 | .011-.02 |
| Selenium | <380-570 | <.38-.57 | 360-680 | .36-.68 | 330-420 | .33-.42 |

**Commented [KL10]:** Inorganic or total

| | Fall Chinook | | Spring Chinook | | Coho | |
|---|---|---|---|---|---|---|
| Contaminant | ug/kg | ppb | ug/kg | ppb | ug/kg | ppb |
| p,p'-DDE | 5-53 | 5-53 | 11-22 | 11-22 | 31-37 | 31-37 |
| p,p'-DDT | <2-7 | <2-7 | 3-8 | 3-8 | <2-4 | <2-4 |
| Arochlor 1254 | 10-47 | 10-47 | 13-26 | 13-26 | 18-19 | 18-19 |
| Arochlor 1260 | <19 | <19 | <18 | <18 | <18 | <18 |

| | Fall Chinook | | Spring Chinook | | Coho | |
|---|---|---|---|---|---|---|
| Contaminant | ug/kg | ppt | ug/kg | ppt | ug/kg | ppt |
| 2,3,7,8-TCDD | <0.0000 - 0.00006 | <0 to .06 | <0.00001 - 0.0001 | <.01-.1 | <0.00001 | <.01 |
| 2,3,7,8-TCDF | 0.00043-0.0014 | .43 – 1.4 | 0.00057 - 0.0011 | .57-1.1 | 0.00036-0.00049 | .36-.49 |

However, large gaps presently exist amongst years that contaminant data were collected for individual fish species through ADEC's Fish Tissue Monitoring Program and the other studies mentioned above, sample sizes and data are limited for particular species (especially sockeye salmon), and previous investigations did not always target whole body fish samples or specific fish organs/portions eaten in traditional subsistence diets. Additionally, an assessment of subsistence consumption rates (fish and shellfish) of Cook Inlet tribal members from Seldovia, Port Graham, Nanwalek, and Tyonek (conducted between 2011 and 2012) undertaken by SVT, revealed that tribal members consume a much larger amount of fish per day (grams/day or g/d), on average, than what is used and/or recommended by state and federal agencies to establish water quality standards in Alaska based on human health criteria (94.8 g/d vs. 6.5 g/d and 17.5 g/d respectively). This implies that contaminants present in the waters of Cook Inlet, and subsequently in the foods eaten in traditional subsistence diets, may be having a much more significant impact on the health of tribal members than previously thought. "Tighter" water quality standards that reduce the amount of contaminants allowed to be discharged into Cook Inlet may be required to protect the health of Cook Inlet tribal members.

**Commented [KL11]:** What statistic is the Seldovia result? The federal values are 90th percentiles of short term dietary recall data including non-fish consumers.

SVT wishes to obtain more current contaminant data for priority subsistence foods of Cook Inlet tribal members given the aforementioned concerns. Based on the assessment completed by SVT in 2012, sockeye salmon was determined to be one of the top fish species eaten by Cook Inlet tribal members. Given the importance of sockeye salmon in traditional native diets and the limited amount of contaminant data available for this species within Cook Inlet, SVT will undertake tissue

**Commented [KL12]:** Though salmon are an undeniably important fish species, some consideration might have been given to selecting species that had smaller home ranges and that would have contaminant body burdens obtained by exposure to local contaminant sources.

Juvenile salmonids might also have been sampled, as their viability is affected by pollutants and their contaminant body burdens are site specific.

It appears that the project wishes to characterize sockeye contaminant concentrations as a function of location and season. These data quality objectives should be presented here. The limited number of samples will likely make it difficult to determine if there are statistically significant differences between different locations or different seasons.

sampling of 36 whole body sockeye salmon (harvested within Cook Inlet) for analysis of several contaminants in the summer of 2014. As evident from the data provided by ADEC fish tissue monitoring program staff, there is a significant gap in contaminant data (especially for organic contaminants) for sockeye salmon within Cook Inlet. To our knowledge and based on the information provided by ADEC, the last contaminant data collected for sockeye salmon within Cook Inlet was between 2002-2003 as part of the ATSDR study Seldovia, Port Graham, Nanwalek, and Tyonek participated in and this was very limited. ADEC's fish tissue monitoring program is supported by funding from EPA, NOAA and BOEMRE (formerly MMS) and the data collected through this program allows ADEC to identify within Alaska waters where: 1) on-going routine sampling is needed for sentinel monitoring, 2) areas or species that may need further evaluation, 3) new species or locations that need to be assessed, and 4) actions needed to be taken to mitigate any negative impacts of environmental pollutants on Alaska's environmental resources. This is a collaborative program and so the data is shared with university researchers, other state and federal agencies (EPA, NOAA, Department of Interior, ADF&G, DHSS) to further work in evaluating toxicologic impacts on coastal ecosystems and salmon health issues. The data collected as a result of SVT's involvement in this project will not only help tribes, state, and federal agencies acquire more data on contaminant levels of sockeye salmon (and fish in general) within Cook Inlet (particularly in regards to organic contaminants), but these important data can be used to examine contaminant levels at regional and global levels amongst salmon populations and to better understand the implications thereof.

This project will be undertaken as a collaborative effort between SVT, the Port Graham Tribal Council, the Nanwalek IRA Council, the Native Village of Tyonek, EPA, and the ADEC. These data will add to pre-existing databases of contaminant concentrations within Cook Inlet. Such databases are important resources when state and federal agencies are considering issuing, updating, or removing human health fish consumption advisories, when undertaking environmental impact assessments, and when establishing or updating water quality standards. The ADEC is currently undergoing their triennial review process for water quality standards in Alaska so these data will be particularly relevant in addition to the fish consumption rates obtained for Cook Inlet tribal members during the recent assessment.

**4.0 DESCRIPTION OF WORK TO BE PERFORMED**

- Collaborate with the ADEC, the Nanwalek IRA Council, the Port Graham Tribal Council, and the Native Village of Tyonek
  - Correspond with partner tribes and ADEC through teleconference calls and e-mail to keep updated with progress. Schedule planning meetings as needed
  - Develop QAPP and send to partner villages, ADEC, and EPA for comments, edits, and approval
  - Develop and post job description to hire samplers from each participating village
  - Hire two samplers who are fishing subsistence nets or have personal use fishing permits, commercial fishing licenses, and/or sport fishing licenses from each village
  - Samplers trained in proper collection and quality control techniques

- SVT Environmental Coordinator and Assistant will travel to each village, during sampling/collection events, to oversee project activities and proper preparation and shipping procedures are followed for transport of fish
- Share findings/results with EPA, ADEC, and partner villages

- Collect 36 whole-body (WB) sockeye salmon within Cook Inlet in the summer of 2014
  - Purchase plastic leak proof/fish bags
  - Collect 3 sockeye salmon specimens from around each participating village (Seldovia, Port Graham, Nanwalek, and Tyonek) during sockeye runs at three different times in the summer of 2014 (at the beginning, at the middle, and towards the end of the run). For each village, per each sampling event, the three fish collected will be analyzed whole-body as a single composite sample. So, 12 composite samples will be analyzed in total although 36 fish will be collected.
  - Upon collection:
    - Fish, will be immediately, and individually, placed into labeled plastic leak proof/fish bags (labeled on outside of bag) using fresh nitrile gloves
    - Once the fish are placed into fish bags, fish bags will immediately be kept on ice in a cooler until arriving back on shore and then frozen at -20°C or -4°F. Thermometers will be provided and used to ensure proper temperature.
    - Fish sampling forms used by ADEC for Fish Tissue Testing Program will be filled out by sampler(s) and included with specimens
    - Labels made out of write in the rain paper will be placed inside bags
  - Provide data that represents expected exposure areas for the target fish species (i.e. within their home range)
  - Provide data that represents areas where target fish species is harvested and consumed from
  - Provide data that can be used to estimate human health and ecological risk from exposure to contaminants in fish
- Prepare and ship fish (within 24 hours) to ADEC's Environmental Health (ADEC EH) Laboratory for analysis following proper procedures
- Once fish have arrived at the ADEC EH Laboratory:
  - All fish will be stored at -20°C or -4°F or below until they are ready to be thawed for processing
  - Fork length, weight, and sex will be recorded
  - All fish will be thawed at 4°C until they are soft enough to be filleted
  - After fish are filleted, fillets are homogenized and the homogenate is then divided into 4 oz sample jars for analysis
  - Otoliths are removed from the fish for age determination
  - For whole body sample analysis, rest of fish is homogenized and put into 4 oz sample jars
  - For each village, the three fish collected per sampling event, will be processed and analyzed whole body for contaminants as a single composite sample
  - All sample jars containing samples are stored at -20°C or -4°F or below until they are analyzed and/or shipped to sub-contracted laboratory (AXYS laboratory (for organic contaminants being tested))
  - All samples analyzed for contaminants (see attached Appendices B, C, E-G)
- Samples will be analyzed for the following contaminants:
  - Polychlorinated biphenyls (PCBs)
  - Chlorinated phenolics (pesticides)
  - Flame-retardant Polybrominated Diphenyl Ethers (PBDEs)

**Commented [KL13]:** More location specific information should be provided if possible.

3 samples will be inadequate to determine the variance in tissue concentrations and compute 95% UCLs on a location specific basis. It will be difficult to determine the significance of seasonal variation on a location specific basis with only one sample. Since Tyonek is so far from the other locations, one might consider adding more samples for Tyonek

**Commented [GG14]:** What will be the target sample size?

**Commented [GG15]:** If the analysis is for whole fish why will the fish be filleted?

**Formatted:** Highlight

**Commented [KL16]:** These appendices need to be provided for review.

**Commented [KL17]:** How much tissue will need to be stored? What analytical reporting limits are needed? What analytical methods will be used?

**Commented [GG18]:** Chlorinated phenolics are chlorinated phenols, guaiacols & vannilins. Are you sure this is what you need. If its pesticides it will be chlorinated pesticides (full list)

- Heavy metals (mercury, arsenic, cadmium, copper, lead, and selenium)
- Conduct data review, evaluation, and analysis
- Prepare reports
- Prepare 1 page success story

**5.0 SAMPLING DESIGN AND METHODS**

The fish collection methods described in this section are intended to provide standardized, reliable, and repeatable results. Additionally, these methods are consistent with methods utilized by ADEC in their Fish Tissue Testing Program and were derived from ADEC.

5.1 Field Crew

SVT's Environmental Assistant will serve as Project Manager and be responsible for carrying out project activities. The Environmental Assistant will report to the Environmental Coordinator. Assurance oversight of grant requirements and project management are responsibilities of SVT's Environmental Coordinator and SVT's President/CEO ensures project compliance to the EPA and other regulatory agencies. In the four villages, two local residents will be hired to collect fish specimens. SVT's Environmental Assistant and Coordinator will travel to each of the partner villages, at least for the 1st collection event, to ensure samplers are following proper procedures and quality assurance/quality control is maintained.

5.2 Field Operations Schedule

Field work described in this QAPP is expected to take place during the summer of 2014. Three whole body (WB) sockeye salmon will be collected from around each participating village (Seldovia, Port Graham, Nanwalek, and Tyonek) during sockeye runs at three different times in the summer of 2014 (at the beginning, at the middle, and towards the end of the run). In total, 36 WB sockeye salmon will be sent to ADEC for analysis of contaminants.

Based upon typical timing runs of sockeye salmon around each village, sampling/collection events for each village are anticipated to take place:

*Seldovia*

1st sampling event (early summer): early June

2nd sampling event (mid summer): mid-July

3rd sampling event (late summer): mid-August

*Port Graham*

1st sampling event (early summer): early June

2nd sampling event (mid summer): mid-July

3rd sampling event (late summer): mid-August

*Nanwalek*

1st sampling event (early summer): early June

2nd sampling event (mid summer): mid-July

3rd sampling event (late summer): mid-August

*Tyonek*

1st sampling event (early summer): early-June

2nd sampling event (mid summer): mid-July

3rd sampling event (late summer): mid-August

Adjustments to sampling dates may be necessary to account for variable conditions such as inclement weather, difficulties in accessing sampling locations, time needed to collect the fish, and sockeye run times.

5.3 Sampling Location Selection Procedure

Fishing sites will be within 100 miles from each village. Based on survey information collected between 2011-2012, the vast majority of community members living in Seldovia, Tyonek, Port Graham, and Nanwalek fish within 25 miles of their respective villages. Sampling locations will be chosen based on local knowledge of where the target fish species can be found and are typically harvested from.

**Commented [KL19]:** This is an improvement over previous text. Are there any sources of contamination to consider? If you want to ascertain the effects of contaminants, you should obtain samples close to the sources of those contaminants. Again, it is unlikely that sockeye salmon adults will have contaminant body burdens affected by local sources. Juvenile salmonids would be better for this purpose.

5.4 Sampling Gear

Gill or set nets with mesh sizes appropriate, and legal, for catching adult sockeye salmon will be used to avoid excessive sampling effort and minimize by-catch of smaller fish. The gill nets and supporting lines will be constructed of non-tarred monofilament or twine to avoid contamination with petroleum-based compounds.

Gear and equipment required for every sampling event is provided in the table below:

| **Equipment and Supply List for Onboard Fish Collection Activities** | |
|---|---|
| Equipment | Minimum Quantity |
| Sampling vessel (including boat, motor, oars, fuel, adequate lighting and required safety equipment) | 1 |
| Gill nets (anchors, depth adjustment lines, and floats) | 1 |
| U.S. Coast Guard-approved personal floatation devices | 4 |
| Maps of sampling areas and sites | 1 |
| Nitrile gloves | 6 pairs |

| | |
|---|---|
| GPS unit | 1 |
| Depth finder | 1 |
| Ice chest | 1 |
| Buckets | 1 |
| Bags of ice | 5 |
| Fish bags | 3 |
| Labels on write in the rain paper | 3 |
| Copy of QAPP | 1 |
| Fish sampling forms | 3 |
| Sharpies, pens, and pencils | 2 of each |
| Fishing licenses/permits | 2 |
| First aid kit | 1 |
| Marine-band radio | 1 |
| Cell phone | 1 |
| Camera | 1 |

5.5 Fish Collection Procedure

From each village, two "samplers" (locally hired) will be responsible for collecting the sockeye salmon from their surrounding fishing area(s). At least one of the samplers hired per village must own, or have access to, a boat and be familiar with how to operate it. Both samplers must have fishing skills, knowledge and have a subsistence net they are fishing or a personal use fishing permit, commercial fishing license, and/or sport fishing license.

**Commented [KL20]:** Reference section 5.3.

SVT's Environmental Coordinator and Assistant will travel to participating villages during sampling events and oversee activities, including accompanying samplers on the boat.

In general, gill nets will be set with local knowledge of when the fish are running and best times to set and pick during the time the Environmental Coordinator and Environmental Assistant are present to help collect the fish. Placement of gill nets at each site will be determined based on the targeted species and the site characteristics. Global positioning system (GPS) coordinates will be recorded for the specific locations where each gill net or trap is deployed. Fish will be immediately removed from the nets as the nets are pulled into the boat, sockeye salmon retained, and nontarget species kept, released, or disposed of in accordance with samplers' fishing licenses/permits and/or any other local fishing regulations they are subject to. For each village, per sampling event, only three sockeye salmon will be shipped to ADEC's laboratory and these specimens will be kept and stored separate from other fish samplers might keep. Samplers will wear fresh nitrile gloves when bagging the fish specimens. Special care will be taken to ensure that petroleum products such as grease or fuel do not come in direct contact with the fish specimens or with surfaces that contact the fish specimens.

Salmon very from stream to stream in length and weight. In Alaska, sockeye salmon vary in length from 18 to 31 inches and weigh between 4 to 15 pounds. Kenai River Reds are much bigger than Nanwalek Reds and Nanwalek Reds are bigger than the Tutka Bay Hatchery Reds. Most people who catch Red salmon can tell the difference in where the fish are from that they are catching. Hatchery fish will also not have a dorsal fin. Hatchery fish will not be sent to ADEC for analysis. Three sockeye salmon will be shipped to the ADEC EH Laboratory as whole body specimens. Only sockeye salmon that are 18" or longer will be used as specimens, thus reducing the likelihood of getting a jack salmon.

**Commented [KL21]:** A fisheries biologist should review this QAPP and how salmon life cycles may affect contaminant body burdens. Juvenile sockeye may reside in freshwater for 1 to 3 years. Adults may return at years 4 and sometimes 5-6. The variation in life history may affect contaminant levels.

**Commented [GG22]:** Will all salmon collected be 18 inches? Or within 25% of 18 inches?

Following is an overview of the fish collection procedures:

1. Transport sample equipment and samplers by boat to sampling locations
2. Deploy and retrieve sampling gear
3. Upon collection, fish will be immediately transferred from sampling gear to fish bags (using nitrile gloves). Fish bags will be kept in a cooler containing ice
4. Prepare and complete field sampling records and documentation that will be enclosed in a zip lock bag and put inside the cooler
5. Prepare labels to put inside fish bags and label outside of bags with permanent marker
6. Return to shore and store specimens (within their bags) in freezer until frozen. Specimens will be stored frozen @ -20°C or -4°F. Thermometers will be provided and used to ensure proper temperature.
7. Ship specimens to laboratory for analysis within 24 hours

**Commented [GG23]:** How will the integrity of the labels maintained if it will be inside the fish bag with the fish. It is recommended that a double bag be used and place the sample label on the outside of first bag then the marker pen on the second bag.

5.6 Labels and Field Documentation

Labels will be made from write in the rain paper and contain:
1) Sample number
2) Sample Date
3) Species
4) Location (lat and long)
5) Site Name
6) Sampler

Labels will be filled out in pencil and placed inside fish bags with specimens.

Additionally, for each collection event, ADEC's fish sampling form will be filled out, enclosed in a zip lock bag, and then placed in the cooler with fish for shipping. Information included in the fish sampling form is as follows:

1) Sample Number
2) Sample Date
3) Species
4) Location (lat and long)
5) Site Name
6) Sampler Affiliation
7) Lead Sampler Signature

A copy of the Fish Sampling Form is provided below.

**FISH SAMPLING FORM**
**ADEC FISH TISSUE TESTING PROGRAM 2013**

| Sample # | Sample Date | Species | Location Lat | Location Long | Site Name | General Area |
|---|---|---|---|---|---|---|
| | | | | | | |
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| notes: | | | | | | |
| | | | | | | |
| | | Sampler Affiliation | | | | Lead Sampler Signature |

**SAMPLING PROTOCOL**

| |
|---|
| Wear fresh gloves to bag each sample in zip-lock bags |
| Label samples consecutively by marking on sample bag |
| Place bagged samples on ice until freezing or shipment |
| Put Sample Collection Form in a zip-lock bag with the samples |
| Check with DEC Environmental Health Lab (howard.teas@alaska.gov, 907-375-8211) for shipping information |
| Take care to ensure that samples aren't exposed to exhaust or gasoline during sample collection |
| Collect as much location information as possible; Lat/long, ADF&G Sub-area, nearest point of land, or general description, as available |
| General Areas: BS (Bering Sea), GOA (Gulf of Alaska), SE (Southeast), Aleutians, Bristol Bay, PWS, Cook Inlet |
| If possible, give latitude and longitude in decimal degrees |

5.7 Storage and Transfer of Fish for Laboratory Analysis

After a collection event, fish specimens will be stored in a cooler (containing ice) and immediately upon returning to shore, the fish bags (containing the specimens) and bags of ice will be immediately placed into a freezer. Specimens will be frozen -20°C or -4°F or below (thermometers will be provided and used to ensure proper temperature) and shipped on ice in a cooler to the ADEC EH Laboratory within 24 hours of the collection date/time. All fish will be checked and identified upon arrival and frozen (-20 C or -4°F). Coolers will be labeled, sealed, and shipped as directed by ADEC staff involved in the Fish Tissue Testing Program.

5.8 Chain-of-Custody Forms

Fish Sampling Forms will serve as Chain-of-Custody Forms (as approved by ADEC) in regards to specimens being transferred from villages to ADEC EH Laboratory. Separate Chain-of-Custody forms will be used for transfer of samples from ADEC EH Laboratory to AXYS laboratory (see Appendix D)

5.9 Laboratory Analysis (see Appendices B, C, E-G)

*Processing*

**Commented [KL24]:** These need to be provided for review.

**Commented [KL25]:** Much better than the last draft.

Samples will be sent to AXYS Analytical Services in Sydney, B.C., for testing of organic contaminants (PCBs, Chlorinated phenolics (pesticides), and PBDEs) and the State of Alaska (ADEC)'s Environmental Health (ADEC EH) Laboratory in Anchorage, Alaska, for testing of heavy metals. However, all specimens will be shipped from villages directly to the ADEC EH Laboratory, whose staff will then ship samples to AXYS accordingly. The chemicals targeted by the ADEC EH Laboratory are: arsenic, cadmium, chromium, lead, selenium, nickel, methylmercury, and total mercury. EPA methods for the chemical analyses are identified in the below tables and attached SOPs (Appendices B, C, E-G). An aliquot of selected homogenized samples will be sent to a contract lab under chain-of-custody for additional chemical analysis. The contract lab (AXYS) will analyze the samples for selected PCB, and dioxin and furan congeners, following EPA approved methods, while percent lipid will be determined gravimetrically, and PBDEs will be analyzed using a modified EPA method (EPA 1668), as there is no standard EPA method for this analyte.

Once the results of the analytical data have been validated, the Alaska Department of Health and Social Services, Section of Epidemiology will evaluate the data from a public health perspective to develop consumption advice and coordinate with ADEC to identify any areas requiring additional sampling.

When specimens are received at the ADEC EH Laboratory, a lab technician will evaluate the fish to ensure that they were properly labeled, packaged and received with sufficient ice to keep them at near-freezing or colder temperature, and that they arrived in good condition. Collection information will be entered into a database and each sample given a unique laboratory identification number. The sample will then be placed in a freezer (temperature range –15 to –20˚C). The laboratory technician will record the physical data for each fish (fork length, weight, sex) and collect the otolith for age determination. If a sample is received that is not in adequate condition e.g. received without ice, decomposed, or otherwise physically damaged to compromise the integrity of the sample, the lab technician will make a record to note the disposal of the sample and SVT staff will be contacted to obtain additional replacement fish samples through coordination with partner villages. The cleaning and preparation of all equipment prior to processing fish tissue will ensure no cross contamination between samples. All knives, cutting boards and grinder parts will be washed with an approved laboratory detergent and rinsed with distilled water. Then all equipment will be rinsed with acetone, hexane, dichloromethane (3 times each) and air dried.

Each fish will be placed on a pre-cleaned cutting board. Boneless, skinless fillets (2), with belly tissue still attached, will be removed from each fish, and homogenized. For whole body analyses, the rest of each fish is homogenized. For each village, per sampling event, the three specimens sent in will be analyzed as one whole body, composite sample. The homogenate will be divided into 4 portions. Each sample portion (one for trace metals, one for all other contaminants, one for reference, one for potential use as a blind duplicate) will be placed in an approved (certified pre-cleaned) glass sample container (I-Chem jar) labeled with the sample number and frozen at -15 to -20˚C. Maximum holding time for frozen samples is one year. The sample portions being sent to the contract lab will be held at the ADEC EH Laboratory until there are a minimum of 15 samples to send. The reference sample will be vacuum-sealed in a 4-mil food grade bag prior to storage in a freezer at the ADEC EH Laboratory. The freezer will be armed with a temperature alarm, and internal temperature in the freezer will be continuously monitored. If the temperature in the freezer moves outside a range of -15 to -20˚C an audible alarm will sound in the lab. If after hours, the lab chief or a designated employee will be notified, and will respond appropriately.

**Commented [GG26]:** Section 4 states that ADEC will process and homogenize whole body samples, then sample aliquots will be bottled for shipment to labs for analysis. The homogenization of the tissue samples must be conducted by the same lab following the same SOP.

**Commented [KL27]:** What is the homogenization process? Equal weights from each fish? Complete homogenization of the fish?

**Commented [GG28]:** Will this be for chlorinated pesticides?

**Commented [GG29]:** EPA Method for PBDE is Method 1614A

**Commented [KL30]:** What comments have these agencies provided on the adequacy of the data for evaluating public health concerns.

**Commented [GG31]:** If the sample will be analyzed whole body- what is this sentence for. Please delete to avoid confusion.

**Commented [KL32]:** If the whole fish is homogenized, a larger fish will have a greater contribution to sample results. If equal weights from each fish are homogenized, then the size of the fish will not be a factor. From a risk assessment perspective, homogenization of the entire fish will produce a concentration term indicative of a human consuming fish from a wild population.

**Commented [GG33]:** A whole body analysis includes the whole body of fish, entrails head and skin on during homogenization. Please send to EPA the homogenization SOP.

At least three grinder rinsate samples will be prepared by washing the grinder following standard procedures, and then rinsing de-ionized water over the grinding surfaces and into amber glass bottles. The rinsate samples will be kept refrigerated and included with the tissue samples in the analysis.

For each group of 10 or fewer samples analyzed at the ADEC EH Laboratory, a duplicate of one of those samples will also be analyzed. For each duplicate a second tissue sample will be removed from one of the jars and analyzed. A random number table will be used to determine which of the samples in the group to use.

For each group of 10 samples or fewer samples sent to the contract lab (AXYS), a blind duplicate sample will be added. It will consist of an aliquot of the homogenate from a fish, giving it a separate sample ID number, and shipping it to the contract lab as a separate sample. The contract lab will provide a frozen sub-sample of each homogenized tissue sample, under chain-of-custody, to the subcontract lab (if necessary). This will include the blind duplicates.

The ADEC EH Laboratory will express ship (overnight delivery) frozen samples to the contract lab (AXYS). The contract lab will be notified via fax or electronically (email) of the impending delivery, along with the tracking numbers. The shipment will include a chain-of-custody document (see Appendix D) and an explanation of what samples were shipped, including identification numbers. If the contract lab does not receive the sample within 24 hours of shipment from the ADEC EH Laboratory, they will contact the Quality Assurance Officer of the ADEC EH Laboratory and report the delay. The ADEC EH Laboratory will also contact the contract lab to confirm receipt of the sample shipment. In the case of delayed receipt the Quality Assurance Officer of the ADEC EH Laboratory will determine if the delay has impacted the integrity of the samples or will affect the quality of the analytical data. If there is any question, the samples will not be analyzed and new samples will be sent. All incidences will be recorded on the chain-of-custody paperwork (see Appendix D).

The contract lab will keep all samples frozen until processed for analysis. All sample material remaining after subsamples are removed for extraction will be refrozen. The contract lab will hold all excess sample material and extracts for one year after the results have been delivered. At that time the contract lab will contact appropriate ADEC EH Laboratory or ADEC personnel to determine whether the samples and extracts are to be discarded or returned to the state. Conditions required for the storage of the samples and extracts at the contract lab are that they be kept frozen at -20˚C and in the dark at all times.

Holding times, for samples to be analyzed, for frozen organic samples is 1 year, extract holding extending another year. For metals the holding time is not well defined but generally 6 months.

*Target Compounds and Detection Limits*

| **Parameter** | **Matrix** | **Minimum Detection Limits at 95% Confidence level (ppm)** |
|---|---|---|
| Total Mercury | Skinless Fillet | 0.005 |
| Cadmium | Skinless Fillet | 0.005 |

**Commented [KL34]:** What levels of concern must be attained from a human health risk perspective? Alternatively, if existing data suggest that a contaminant will always be detected, it is not necessary to specify human health risk based reporting limits.

| | | |
|---|---|---|
| Copper | Skinless Fillet | 0.2 |
| Lead | Skinless Fillet | 0.02 |
| Selenium | Skinless Fillet | Undetermined at this time |
| Total arsenic | Skinless Fillet | 0.05 |
| Organochlorine Pesticides | Skinless Fillet | See Appendix B |
| PCB Congeners | Skinless Fillet | See Appendix B |
| PBDE Congeners | Skinless Fillet | See Appendix B |
| Percent Lipid | Skinless Fillet | |

[1] – FDA Action/Guidance Levels only exist for Crustacea and Molluscs.

**Commented [GG35]:** Why skinless fillets – I thought whole body salmon will be analyzed? Please verify.

1. Percent Lipid

The determination of lipid content in a sample extract is carried out by quantitatively measuring (by weight or by volume) an aliquot of an extract prepared for one of the organic analyses to be performed on the samples, typically either the PCB or dioxin/furan analysis. Each aliquot is placed into a pre-weighed foil weigh boat. The solvent is allowed to evaporate at room temperature prior to drying of the extract at 105°C for 30 minutes. When cool, the weigh boat is re-weighed to determine the weight of lipid. The percent lipid in the sub-sample of extract is determined as the weight of the remaining material divided by the weight of the sample with solvent. The above lipid determination is performed in duplicate and the average percent lipid is reported. The percent recoveries of the labeled surrogate compounds in the remaining extract are corrected for amount of extract consumed in the lipid determination.

2. Trace Metals

Analysis for Arsenic, Cadmium, Chromium, Lead, Selenium and Nickel will be performed by the following method(s). The most recent revision of each method as listed in SW-846 will be used:

| Analyte | Preparatory Method | Analytical Method |
|---|---|---|
| Arsenic | EPA Method 3050, 3051 or 3052 | EPA Method 7060 or 6020 |
| Cadmium | EPA Method 3050, 3051 or 3052 | EPA Method 7131 or 6020 |
| Copper | EPA Method 3050, 3051 or 3052 | EPA Method 7191 or 6020 |
| Lead | EPA Method 3050, 3051 or 3052 | EPA Method 7421 or 6020 |
| Selenium | EPA Method 3050, 3051 or 3052 | EPA Method 7421 or 6020 |

**Commented [GG36]:** We need the target reporting limits for all the parameters for this project. Also needed are the MDLs/MRLS of the laboratories that will analyze the tissue samples.

**Commented [KL37]:** Are these methods the same as those used to collect existing data? If not, will results be comparable?

**Commented [GG38]:** Recommends not to use GFAA for analysis – Use method 6010 or 6020 – Both ICP methods.

**Commented [KL39]:** I believe these will provide total arsenic results. Some consideration should be given to using 1632a, which will detect inorganic arsenic, the arsenic form which is of primary toxicologic concern.

The preferred method for metals analysis for fish tissues and other environmental matrices is EPA Method 6020, "*Inductively Coupled Plasma - Mass Spectrometry.* The use of ICP/MS technology will enable the laboratory to measure the presence of metals in seafood at the lowest possible levels with greater efficiency and savings. The same measurement quality objectives for Trace Metals Analysis as listed in the below tables will be followed.

3. Total Mercury

Total mercury will be determined by EPA Method 7473, Mercury in Solids and Solutions by Thermal Decomposition, Amalgamation, and Atomic Absorption Spectrometry.

4. Organochlorine Pesticides
Organochlorine pesticides will be determined by USEPA Method 8081 or equivalent method.

5. PCB and PBDE Congeners
Analysis for Polychlorinated Biphenyls (PCB) and Polybrominated Diphenyl Ethers (PBDE) congeners will be determined by USEPA Method 1668A. The cleanup techniques described in the method will be employed as necessary to eliminate interferences and to obtain the best possible reporting limits.

**Commented [GG40]:** Will the samples be analyzed for a full list of 209 PCB congeners by 1668A and also a full list of PBDEs using Method 1614 with extract spilt form 1668A?

**6.0 QUALITY OBJECTIVES AND CRITERIA**

6.1 Project Quality Objectives

In order for the data collected in this project to be most effectively utilized by SVT, other tribes, and state and federal agencies, knowledge of the following components is required:

- Present and historical sources of contaminants within Cook Inlet
- Planned development and industries within Cook Inlet that potentially will result in future sources of contaminants
- Nature and extent of current, and historical, contaminants in fish and shellfish within Cook Inlet, throughout the state of Alaska, and in systems with similar types, and levels, of development and industries
- Patterns of fish use and consumption by tribal consumers following traditional subsistence lifestyles
- Ecological receptors and exposure pathways for contaminated fish tissue

**Commented [KL41]:** This consideration should be evaluated with regards to sample location. If contaminants are in fish, it would make sense to take samples in proximity to areas of influence of contaminant sources.

The primary purpose of this project is to gather data to support human and ecological risk assessments for Cook Inlet. The data collected as part of this project may have value in:

**Commented [KL42]:** It appears that the project is looking at variation over time.

- Characterizing spatial patterns of contaminants
- Correlating tissue concentrations with contaminant concentrations in sediment
- Comparing contaminant levels among fish species
- Comparing contaminant levels among river reaches
- Characterizing the variation in contaminant concentrations within fish of a single species (sockeye salmon)
- Comparing contaminant data collected in 2014 to data collected in other years

**Commented [GG43]:** There is no mention of a collocated sediment collection with the fish – so how can a correlation be done?

It is also unlikely that sediment contaminant concentrations will impact the body burden of returning adult salmon. Effects however might be associated with spawning success of adults or viability of juvenile salmonids or the viability of eggs.

**Commented [KL44]:** Expand on this. The locations where fish are being collected are quite broad. How are sampling locations to be tied to river reaches?

**Commented [KL45]:** Again, the impact of the life history of sockeye on contaminant concentrations should be considered.

Since collection methods will follow those established by ADEC's Fish Tissue Testing Program and this is a collaborative project with ADEC, project results will be standardized; incorporated into ADEC's databases and on-going research; and shared with university researchers, other state and federal agencies (EPA, NOAA, Department of Interior, ADF&G, DHSS) to further work in evaluating toxicologic impacts on the coastal ecosystem and salmon health issues.

Quality Control methods that will be in place during field collection:

1) Copy of QAPP on board boat

2) Use of labels and labeling
3) Use of Fish Sampling Forms
4) SVT Environmental Coordinator and Assistant serving as QA monitors onsite (for at least the 1st collection event at each village)
5) Fish specimens being immediately "bagged" using nitrile gloves and put into a cooler (with ice) while on board boat
6) Fish specimens being kept away from petroleum products/boat exhaust
7) Once collected, fish specimens are taken immediately back to shore and frozen at -20°C or –4°F before being shipped. Thermometers will be provided and used to ensure proper temperature.
8) Specimens shipped within 24 hours

By following the methodology outlined in this QAPP for collecting fish tissue samples, we can be assured of providing high quality samples to the State for useful and defensible information.

## 6.2 Data Quality Objectives

**Commented [KL46]:** What levels of concern must be attained from a human health risk perspective? Alternatively, if existing data suggest that a contaminant will always be detected, it is not necessary to specify human health risk based reporting limits.

Since the specimens will be sent to laboratories utilized and/or owned by ADEC, the Quality Assurance (QA) procedures and analytical SOPs and associated laboratory Quality Control (QC) in terms of types & frequencies of QC samples and QC acceptance limits have been determined to be adequate to meet the data quality needs of this project. The analytical methods used by the two laboratories will be EPA Methods or Standard Methods, both well-documented and published methods (see attached Appendices B, C, E-G). The laboratory-established control limits shall be used as acceptance limits for accuracy and precision for this project. The precision and accuracy control limits are listed in the SOPs for the procedures to be used.

AXYS Analytical Services' quality policies meet or exceed ISO 17025 standards. ISO/IEC 17025:2005 "General Requirements for the Competence of Testing and Calibration Laboratories" is an international standard that specifies the management and technical requirements for competence to perform test measurements and calibrations. The ADEC EH Laboratory certifies commercial and municipal laboratories within the State of Alaska to conduct analyses of drinking water and accredits commercial laboratories to conduct analyses including soil remediation.

As such, the laboratories' QC and SOPs (attached as Appendices A, B, C, E, F, G) have been accepted as the project's measurement performance criteria for the analytical component. The laboratories will report detection levels on a sample/analyte-specific basis. Method detection limits (MDLs) will be provided. A copy of the ADEC Environmental Health (ADEC EH) Laboratory's Quality Manual is attached to this QAPP as Appendix A.

**Summary of Measurement quality objectives for Trace Metals Analysis**

| QC Element | Description | Frequency | Acceptance Criteria | Corrective Action |
|---|---|---|---|---|
| Initial Calibration | ICP: 1 std and blank<br><br>GFAA: 3 stds and blank | Daily | <br><br>r > 0.995 | Reanalyze calibration. |

| | | | | |
|---|---|---|---|---|
| Instrument precision | ICP: %RSD 3 integrations<br><br>GFAA: RPD of 2 injections | Each calibration and calibration verification | ICP: %RSD < 5%<br><br>GFAA: RPD±10% | Recalibrate and reanalyze |
| Initial Calibration Verification (ICV) | Midlevel ($2^{nd}$ source) verification | After initial calibration. | %Rec= ±10% | Recalibrate and reanalyze. |
| Initial Calibration Blank (ICB) | Interference free matrix to assess analysis contamination. | After initial calibration | All analytes < MDL | Re-calibrate and re-analyze |
| Continuing Calibration verification (CCV) | Midlevel verification | Every 10 samples and at end of analytical sequence. | ICP: %Rec= ±10%<br><br>GFAA: %Rec= ±20% | Recalibrate and reanalyze samples not bracketed by acceptable CCV |
| Continuing Calibration Blank (CCB) | Interference free matrix to assess contamination. | Every 10 samples and at end of analytical sequence. | Analytes < MDL | Reanalyze samples not bracketed by an acceptable CCB |
| Method Blank (MB) | Interference free matrix taken through entire preparation and analysis process | One per batch, not to exceed 20 samples per batch. | All target analytes must be < one-half of the Reporting Limit (RL). | If blank $\underline{\geq}$ one half RL, and all samples ND, no action necessary. If blank < 5% of sample results qualify data. If Blank $\underline{\geq}$ one half RL and > 5% of sample results re-extract and re-analyze samples. |
| Laboratory Control Sample (LCS) | Interference free matrix spiked with all target compounds at Midlevel of ICAL | One per batch, not to exceed 20 samples per batch. | %Rec= 80%-120% | If LCS > upper control limit and samples ND, no action needed. Otherwise, re-extract and re-analyze batch. |
| Matrix Spike/ matrix spike duplicate (MS/MSD) | Spike at mid point of ICAL | One MS/MSD per batch, not to exceed 20 samples per batch | Advisory limits<br><br>%Rec= 75%-125%<br>RPD < 25%<br>Evaluate only if spike amount is > 5x sample concentration. | Qualify results. If LCS acceptable and MS/MSD outside limits qualify results as possible matrix effect. |
| Duplicate (Dup) | Laboratory duplicate | One Dup per batch, not to exceed 20 samples per batch | %RPD < 25% | Evaluate results, qualify data. |

| | | | | |
|---|---|---|---|---|
| Post digestion spike (PDA) | Sample digestate spiked with target analytes | ICP: 1 per batch.<br><br>GFAA: Every sample | %Rec=75%-125%. | Re-extract and/or re-analyze sample(s). |
| Serial dilution | 1:4 dilution analyzed to assess matrix effects | As needed to assess new matrices. | Agreement between undiluted and diluted results ±10% | Evaluate data, may require dilution and reanalysis of samples. |
| Method of Standard Additions (MSA) | Method of quantitation | As needed for samples with suspected or confirmed matrix effects | r ≥ 0.995 | Evaluate data. |
| Method Detection Limit Study (MDL) | Minimum 7 replicates spiked at 3-5 times the estimated MDL | Prior to analysis of samples, then annually | 40 CFR Part 136, Appendix B | Acceptable MDL must be performed prior to sample analysis. |
| Initial Demonstration of Capability (IDC) | 4 replicates of LCS | Prior to analysis of samples, then annually | %Rec= 80%-120% | Re-extract and re-analyze IDC. Acceptable IDC must be performed prior to analysis of samples. |
| Performance Evaluation (PE) | Single blind, standard reference material, outside vendor or agency. | Prior to analysis of samples, then annually | Acceptance limits established by PE sample vendor or agency | Determine cause of error. Re-analyze new PE sample. Acceptable PE must be performed prior to analysis of samples. |

**Summary of Measurement quality objectives for Mercury by EPA Method 7473**

| QC Element | Description | Frequency | Acceptance Criteria | Corrective Action |
|---|---|---|---|---|
| Initial Calibration (ICAL) | Low range: 4 standards and blank<br><br>High range: 6 standards and blank | Prior to sample analysis, then as required | r > 0.995 | Reanalyze calibration. |
| Initial Calibration Verification (ICV) | Midlevel (2nd source) verification. Includes at least a high and low concentration standard for each working range. | After initial calibration | %Rec= ±10% | Recalibrate |
| Daily Calibration | Midlevel verification includes at least a high and low concentration standard for each working range. | Daily, after every 10 samples and at end of analytical sequence. | %Rec= ±10% | Recalibrate and reanalyze samples not bracketed by acceptable Daily Calibration |

| | | | | |
|---|---|---|---|---|
| Method Blank (MB) | Interference free matrix taken through entire preparation and analysis process | One per batch, not to exceed 20 samples per batch. | All target analytes must be < one-half of the Reporting Limit (RL). | If blank ≤ one half RL, and all samples ND, no action necessary. If blank is > 5% of sample results qualify data. If blank ≥ one half RL and > 5% of sample results re-extract and re-analyze samples. |
| Laboratory Control Sample (LCS) | Interference free matrix spiked with all target compounds at Midlevel of ICAL | One per batch, not to exceed 20 samples per batch. | %Rec= 80%-120% | If LCS > upper control limit and samples ND, no action needed. Otherwise, re-extract and re-analyze batch. |
| Matrix Spike/ matrix spike duplicate (MS/MSD) | Spike at mid point of ICAL | One MS/MSD per batch, not to exceed 20 samples per batch | Advisory limits<br><br>%Rec= 80%-120%<br>RPD < 20%<br>Evaluate only if spike amount is > 5x sample concentration. | Qualify results. If LCS acceptable and MS/MSD outside limits qualify results as possible matrix effect. |
| Duplicate (Dup) | Laboratory duplicate | One Dup per 10 samples or fraction of 10 when the batch is greater than 10 | %RPD < 25% | Evaluate results, qualify data. |
| Method of Standard Additions (MSA) | Method of quantitation | As needed for samples with suspected or confirmed matrix effects | r ≥ 0.995 | Evaluate data. |
| Method Detection Limit Study (MDL) | Minimum 7 replicates spiked at 3-5 times the estimated MDL | Prior to analysis of samples, then annually | 40 CFR Part 136, Appendix B | Acceptable MDL must be performed prior to sample analysis. |
| Initial Demonstration of Capability (IDC) | 4 replicates of LCS | Prior to analysis of samples, then annually | %Rec= 80%-120% | Re-extract and re-analyze IDC. Acceptable IDC must be performed prior to analysis of samples. |
| Performance Evaluation (PE) | Single blind, standard reference material, outside vendor or agency. | Prior to analysis of samples, then annually | Acceptance limits established by PE sample vendor or agency | Determine cause of error. Re-analyze new PE sample. Acceptable PE must be performed prior to analysis of samples. |

| **Summary of Measurement quality objectives for USEPA Method 8081** | | | | |
|---|---|---|---|---|
| **QC Element** | **Description** | **Frequency** | **Acceptance Criteria** | **Corrective Action** |

| | | | | |
|---|---|---|---|---|
| Initial Calibration (ICAL) | Minimum 5 points, low level at or below reporting limit | Prior to sample analysis | r $\geq$ 0.995 or $r^2 \geq 0.990$ or %RSD $\leq$ 20% | Recalibrate |
| Initial Calibration Verification | Midlevel (2nd source) verification | After each new ICAL. | %Rec= 85%-115% | Check for problems with ICAL and or second source. Reanalyze ICAL. |
| Continuing Calibration Verification | Midlevel verification | At beginning and end of each 12 hour analytical shift; after every 10 samples and at the end of the analytical sequence whichever is most frequent. | %Drift = 15%, or %D < 15% | Check for problems; reanalyze CCV. If unacceptable after 2nd injection recalibrate. Samples not bracketed with an acceptable Calibration verification must be reanalyzed. |
| Method Blank (MB) | Interference free matrix taken through entire preparation and analysis process | One per batch, not to exceed 20 samples per batch. | All target analytes must be < one-half of the Reporting Limit (RL) | If blank $\geq$ one half RL, and all samples ND, no action necessary. If blank < 5% of sample results qualify data. If Blank $\geq$ one half RL and > 5% of sample results re-extract and re-analyze samples. |
| Laboratory Control Sample (LCS) | Interference free matrix spiked with all target compounds at Midlevel of ICAL | One per batch, not to exceed 20 samples per batch. | %Rec= 50%-130% or in house generated limits. | If LCS > upper control limit and samples ND, no action needed. Otherwise, re-extract and re-analyze batch. |
| Matrix Spike/ matrix spike duplicate (MS/MSD) | Field sample spiked with all target compounds at Midlevel of ICAL | One MS/MSD per batch, not to exceed 20 samples per batch | Advisory limits: %Rec= 50%-130% or in house generated limits.<br><br>RPD < 50%<br>Evaluate only if spike amount is > 5x sample concentration. | Qualify results. If LCS acceptable and MS/MSD outside limits qualify results as possible matrix effect. |
| Duplicate (Dup) | Laboratory duplicate | One Dup per batch, not to exceed 20 samples per batch | %RPD < 50% | Evaluate results, qualify data. |
| Surrogate spikes (Surr) | | Every MB, LCS, MS, MSD, Dup, and sample | %Rec=50%-130% or in house generated limits. | If %Rec is outside limits for MB or LCS re-extract and re-analyze entire batch. If %Rec > upper control limit in a sample and sample is ND for all compounds, |

| | | | | |
|---|---|---|---|---|
| | | | | no action. Otherwise, re-extract and re-analyze samples with surrogate outside limits. |
| Method Detection Limit Study (MDL) | Minimum 7 replicates spiked at 3-5 times the estimated MDL | Prior to analysis of samples, then annually | 40 CFR Part 136, Appendix B | Acceptable MDL must be performed prior to sample analysis. |
| Initial Demonstration of Capability (IDC) | 4 replicates of LCS | Prior to analysis of samples, then annually | %Rec= 50%-130% or in house generated limits. | Re-extract and re-analyze IDC. Acceptable IDC must be performed prior to analysis of samples. |
| Performance Evaluation (PE) | Single blind, standard reference material, outside vendor or agency. | Prior to analysis of samples, then annually | Acceptance limits established by PE sample vendor or agency | Determine cause of error. Re-analyze new PE sample. Acceptable PE must be performed prior to analysis of samples. |

**Summary of Measurement quality objectives for USEPA Method 1668B***

| QC Element | Description | Frequency | Acceptance Criteria | Corrective Action |
|---|---|---|---|---|
| Initial Calibration (ICAL) | Minimum 5 points | Prior to sample analysis | Per method 1668A | Recalibrate |
| Calibration Verification | Midlevel verification | At beginning and end of each 12 hour analytical shift | Per method 1668A | Per method 1668A |
| Method Blank (MB) | Interference free reference matrix taken through entire preparation and analysis process | One per batch, not to exceed 20 samples per batch. | Per method 1668A | Per method 1668A |
| Ongoing Precision and Recovery Sample (OPR) | Interference free reference matrix spiked with all target compounds at Midlevel of ICAL | One per batch, not to exceed 20 samples per batch. | Per method 1668A | Per method 1668A |
| Duplicate (Dup) | Laboratory duplicate | One Dup per batch, not to exceed 20 samples per batch | %RPD < 50% | Evaluate results, qualify data. |
| Surrogate spikes (Surr) | | Every sample and QC sample. | Per method 1668A | Per method 1668A |
| Method Detection Limit Study (MDL) | Minimum 7 replicates spiked at 3-5 times the estimated MDL | Prior to analysis of samples, then annually | 40 CFR Part 136, Appendix B | Acceptable MDL must be performed prior to sample analysis. |

| | | | | |
|---|---|---|---|---|
| Initial Precision and Recovery (IPR) | 4 replicates | Prior to analysis of samples, then annually | Per method 1668A | Re-extract and re-analyze IPR. Acceptable IPR must be performed prior to analysis of samples. |
| Performance Evaluation (PE) | Single blind, standard reference material, outside vendor or agency. | Prior to analysis of samples, then annually | Acceptance limits established by PE sample vendor or agency | Determine cause of error. Re-analyze new PE sample. Acceptable PE must be performed prior to analysis of samples. |

*Additional Quality Control criteria are included in the analytical method.

## 7.0 DELIVERABLES, DATA STORAGE AND ANALYSIS

Once the fish and completed sample data forms are received at the ADEC EH Laboratory, a lab technician will enter the data into a SQL Server database. A unique sample tracking number will be assigned to each sample at that time. Sample collection data sheets, chain-of-custody forms, sample tracking forms, and bench sheets containing calculations will also be filed at the ADEC EH Laboratory. As analyses are performed, raw or calculated results will be added to the database (this will depend on machine output). When subsamples are sent to the contract lab (AXYS), a chain-of-custody form (see Appendix D) will be sent with it, providing a tracking number for the sample. The contract lab will send an electronic copy of the results (a read only file) of all of its analyses back to the ADEC EH Laboratory, with all data referenced by the sample tracking number. A second copy of the results will be sent to a third party contractor for data validation, results of which will be provided as both electronic (read only file) and hard copy files. The electronic data files from the contract lab and the data validation contractor will be downloaded directly into the ADEC EH Laboratory database. The validated data will be used to generate the program reports. Once the data has been validated, a hard copy and electronic copy (as a read only file) will be sent to both EPA Region 10 (as part of the deliverables) and to Seldovia Village Tribe staff, who will then share the data with partner tribes (Port Graham, Tyonek, and Nanwalek). Throughout the study the ADEC EH Laboratory Quality Assurance Officer and ADEC Fish Tissue Program Coordinator will check fish sample processing and the dataset for errors by comparing sample processing and data entry sheets with the results in the electronic database.

**Commented [KL47]:** What coding approach will be used to develop a sample number?

Hard copy documents to be retained at the ADEC EH Laboratory include original chain-of-custody documents (this includes log information), sample metadata (age, sex, length, weight, any abnormalities), in-lab sample tracking forms, sample analytical results of the required analyses, and graphic results of the analyses; the electronic data include sample log-in, data generated during analysis, quality control data, and the final analytical data.

Data deliverables required from all laboratories (to be used for data validation) includes sample analytical results, blind and laboratory duplicates, MS/MSDs blanks, and calibration checks, in addition to the required storage of the electronic raw data generated in both the ADEC EH Laboratory and the contract lab. None of the data will be purged by the labs without prior authorization from the ADEC EH Director.

Data, once received by SVT staff, will be kept on SVT office computers and combined data entered into an excel database (if necessary). SVT offices and computers are secured.

*Data Analysis*

The basic data analysis will be performed with a commercial statistical software program (SPSS Inc.) by ADEC staff. Mean, standard deviation, and median values will be calculated for contaminant concentrations in each species sampled. If sufficient data is available, mean, standard deviation, and median values will also be calculated for groupings within a species: sex, age length, and weight. Analysis of variance and analysis of co-variance may be calculated for contaminant loads among general collection locations for species where there are sufficient data points and the basic assumptions for data quality are met. If the Data quality assumptions are not met, non-parametric alternatives will be used in the analysis. To determine whether there are sufficient data collected for individual analyses, sample size requirements will be calculated, again with SPSS Inc. software.

**Commented [KL48]:** Why not compute sample size requirements **BEFORE** the work is done? A statistician can develop required sample number for different objectives if existing data are available to provide information on variance in salmon contaminant tissue concentrations. Again, the proposed sample numbers are quite low for determining significant differences between locations and across seasons. The life cycle of sockeye salmon should be considered as the age of outmigration and age at return for spawning will likely affect variance in salmon tissue concentrations.

## 8.0 REPORTING AND OUTREACH

Laboratory results will be sent directly to ADEC staff involved in the Fish Tissue Testing Program, who will then compile and analyze those results. Electronic files containing results will be sent to SVT from ADEC. Upon receiving results, SVT will share the results with all partner Tribes, the Tribal Council, and EPA. A project summary report will be developed by SVT Environmental Personnel and sent out to all the above parties as well.

Additionally, SVT's Environmental Coordinator will submit quarterly reports to our IGAP Project Officer to keep EPA informed of project progress.

**Commented [GG49]:** The sections on Assessment and Response and Data Validation and Usability are missing. Please add to the QAPP. Also, identify who will be responsible for validating the analytical data.

## 9.0 REFERENCES

[ADEC] Alaska Department of Environmental Conservation Fish Monitoring program. Available online at http://www.dec.state.ak.us/eh/vet/fish.htm

[ATSDR] Agency for Toxic Substances and Disease Registry. 2009. Evaluation of seafood and plant data collected from Cook Inlet near the native villages of Port Graham, Nanwalek, Seldovia, and Tyonek, Alaska. Atlanta, Georgia

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Assessment of contaminant body burdens and histopathology of fish and shellfish species frequently used for subsistence food by Alaskan Native communities. Available online at http://doc.nprb.org/web/10_prjs/1019_Final%20report.pdf